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COMPLETE SPECIFICATION.

Process for Recovering Saponins from Agave Leaves.

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The present invention concerns the recovery of commercially valuable materials from the non-fibrous components of the leaves of agave plants.

Now and again it has been reported in literature that the non-fibrous components of the agave leaves, i.e. the juice and the so-called flesh, contain a number of commercially valuable materials such as, for example, saponins, and attempts have been made to recover these materials from the agave on an industrial scale. The difficulties, however, which have been encountered in the past in the isolation of commercially utilizable products from the non-fibrous components of the agave leaves have rendered the recovered products too expensive for practical purposes.

It has now been found that under suitable conditions the non-fibrous components of the agave leaves may be separated into two fractions of different properties. One of these is predominantly soluble in non-polar solvents and comprises predominantly lipoidic substances as well as some minerals. This fraction will be referred to hereinafter as the "lipoidic fraction". The other fraction is soluble in lower aliphatic alcohols and will be referred to hereinafter as the saponinic fraction. The lipoidic fraction can be used, for example, as a feed additive for poultry while the saponinic fraction may be used as a surface active agent, or, if desired, can be further worked up for the recovery of some pharmaceutically valuable sapogenins such as, for example, hecogenin.

In accordance with the invention a process for the recovery of saponins and sapogenins from the non-fibrous matter of agave leaves and the simultaneous recovery of non-saponic organic and mineral matter generally referred to herein as "lipoidic fraction" comprises producing from said non-fibrous matter an extract with an aliphatic alcohol having not more than 6 carbon atoms in its molecule, or a mixture of organic solvents containing at least one such alcohol, recovering at least a part of the lipoidic fraction from the alcohol extract and thereafter recovering saponins and sapogenins therefrom.

Thus, in accordance with the invention the saponins contained in the agave leaves are separated from the lipoidic and mineral components thereof.

The saponins and sapogenins, if any, can be recovered from the alcoholic solution by precipitation which may be induced by concentration and/or cooling and/or the addition of non-solvents. The presence or absence of sapogenins in the alcoholic extract depends on whether prior and/or during the extraction the conditions are favourable for the hydrolysis of the saponins.

The starting material for the extraction may be juice, possibly diluted with water as a result of previous operations in which the original leaves are crushed and the sisal fibres which are removed and rinsed with water for cleaning. A mixture of juice and flesh, or a product obtained by the dehydration of such mixture may also be used.

[*Price 4s. 6d.*]

Price 5s. 0d.

Finally, wet or dried juice and/or flesh may also serve as starting material.

Where in the process according to the invention the juice expressed from the agave leaves serves as starting material, the process may comprise the step of concentrating the juice down to a dry-matter content of 50—70% and the remaining syrup is then submitted to extraction with an alcohol. Any lipoidic fraction precipitating during this concentration is separated.

Where dry matter obtained by drying of the juice and/or the flesh of the leaves is used as starting material for the extraction, it is possible to use as extractant an azeotropic mixture of benzene and methanol.

The isolation of the lipoidic and mineral components may be effected simultaneously with or follow the isolation of the saponins as will become apparent from the following description.

The lipoidic fraction obtained in accordance with the invention, i.e. the mixture left after the isolation of the saponins, is not uniform. In addition to various lipoids it contains *inter alia* various minerals, chlorophyll, proteins and carotenoids. It is a particular advantage of the present invention that this mixture is commercially utilizable in the form in which it is obtained, without any purification steps being necessary. As pointed out above, the main use of this mixture is as feed additive for poultry but other applications are also conceivable.

The operation steps required for the separation of the startng material into the two fractions outlined above may vary in accordance with the aggregate state, consistency and concentration of the starting material.

Where the starting material is an aqueous diluate of the original agave juice the first step in the process according to the invention is as a rule concentration in order to produce a concentrate which can readily be extracted with an aliphatic alcohol of not more than 6 C atoms. This concentrate may either be a syrup of 60—70% by weight of dry matter content or dry matter obtained by the complete evaporation of the water. For this concentration it is possible to use either the dilute juice in its original form or, alternatively, to let the juice stand for some time whereupon it separates into a slurry and a supernatant clear solution. The latter is then decanted while the former is used as starting material in the process according to the invention. It should, however, be noted that the above slurry may be extracted directly with the alcohol with or without further concentration.

The concentration proper of the juice or the slurry, as the case may be, is preferably effected in vacuo at a *p*H of from 4 to 8. Since the native juice is as a rule more acidic the concentration is preferably preceded by partial neutralization which is as a rule effected by the addition of solid mild bases such as borax, or calcium hydroxide.

During the concentration, in particular when this is done at atmospheric or only slightly reduced pressure so that the juice has to be heated to or nearly to 100° C., there precipitates from the mother liquor some solid material amounting from 1.5 to 4.5% by weight of the original juice and containing *inter alia* 0.08 to 0.30% of phosphorus (calculated as elementary phosphorus), 0.12 to 0.70% by weight of chlorophyll, 3—12% by weight of proteins, 0.035 to 0.11% by weight of carotenoids, 0.0025 to 0.10% by weight of β-carotene, 1.0 to 4.0% by weight of lipoids and 6.5 to 23.0% by weight of minerals calculated as ash and containing 35 to 45% by weight of calcium oxide and 2 to 3% by weght of magnesium oxide. This precipitate is as a rule isolated as it forms, in particular when the juice is only concentrated down to a syrup, and forms a valuable product of the recovery process according to the invention, which may be used as feed additive for chicken.

Where the syrupy juice obtained after concentration is used as starting material for the extraction the amount of extractant added should preferably be such that its concentration in the resulting mixture is about 70% by volume. The alcoholic extract obtained in this manner may then be re-extracted preferably at slightly elevated temperature with an organic non-polar solvent such as, for example, petroleum ether, benzene, toluene or the like. By this re-extraction the bulk of the fats and pigments accompanying the saponins are removed. These materials may be recovered from the resulting non-polar solvent extract by evaporation of the solvent and be added to lipoidic fractions recovered in previous and/or subsequent steps.

Instead of re-extracting the alcoholic extract with a non-polar organic solvent it is also possible to purify the alcoholic extract by filtration and/or centrifugation thereby recovering any entrained, undissolved matter. This is followed by partial evaporation of the solvent or rather of an azetropic alcohol-water mixture. Evaporation is preferably effected in vacuo in order to prevent or reduce as much as possible any hydrolysis of the saponins. During this evaporation some more non-saponinic material precipitates and is again separated by filtration or centrifugation and combined wth the other lipoidic fractions. Some more lipoidic material is then removed from the alcoholic extract by the addition of some non-polar solvent in the ratio of extract: non-polar solvent of 1 : 1 or 1 : 2 and after removal of the precipitate obtained in this

manner, which latter again consists of lipoidic end products of the process, the filtrate contains mainly saponins. To this filtrate water is added until a phase separation occurs. The non-polar phase is separated from the polar one and the latter is, if desired, again extracted with a non-polar solvent for further purification. The various non-polar fractions are combined and after removal of the solvent some more lipoidic end product is obtained.

After purification of the alcoholic extract in either of the two ways outlined above a solution is obtained containing substantially only saponins. For the recovery of the saponins from the solution water has first to be removed as far as possible. This is equally done by evaporation since the aliphatic alcohol serving as solvent and the water co-extracted with the saponins and/or added at a later stage of purification is removed together with the alcohol in the form of an azetropic mixture. From the concentrated, water-free alcoholic solution obtained in this way there first precipitates a mixture of impurities such as poly-saccharides and mineral salts. These are removed by centrifugation or filtration and the remaining mother liquor is then cooled whereupon the saponins precipitate. In lieu of or in addition to cooling, precipitation may also be induced by the addition of non-solvents, e.g. acetone.

Where the starting material in the process according to the invention is dry material obtained by complete drying of the juice and/or the flesh, this first step in the process according to the invention is extraction of the starting material with an aliphatic alcohol having in its molecule not more than six carbon atoms or with a mixture of such alcohols. Working-up of the alcoholic extract obtained in this manner is then effected in a similar manner as the working-up of the alcoholic extract obtained in the embodiment described above in which juice serves as starting material.

However, in this case extraction with alcohol is not preceded by the removal of a lipoidic fraction as is the case when the juice is concentrated down to a syrup. Accordingly a substantial portion of the lipoidic fraction will be co-extracted by the alcohol. Contrary to what may have been expected, the materials forming this fraction remain in solution and are not precipitated together with the saponins. They may accordingly be recovered at a later stage by admixing water to the filtrate obtained after a precipitation of the saponins with the aid of a non-polar solvent, in an amount sufficient to induce phase separation. The lipoids pass into the non-polar phase while the residual saponins, if any, remain in the aqueous-alcoholic layer. Where the saponins are precipitated by the addition of acetone, the lipoids are recovered from the mother liquor by the simultaneous addition of ligroin or petroleum ether or a similar non-polar organic solvent and of water until phase separation occurs. In this case any sapogenins and saponins which are still present will either remain in the water-alcohol-acetone layer or precipitate therefrom while the lipoids are extracted into the non-polar layer. From the water-alcohol-acetone layer the saponins and sapogenins may then be recovered by complete evaporation of the solvents. Likewise, the lipoids extracted from the alcoholic mother liquor are recovered from their solution by the evaporation of the solvent. The residue may be combined with any lipoidic fraction obtained at an earlier stage.

It is thus seen that irrespective of the aggregational form, concentration and consistency of the starting material, the process according to the invention always comprises the following essential features:—

(a) the starting materials are non-fibrous components of agave leaves;

(b) an alcoholic extract is prepared from the starting material, the alcohol serving as extractant having not more than 6 carbon atoms in its molecule;

(c) the above alcoholic extract is worked up in order to precipitate therefrom saponins and sapogenins;

(d) the lipoids and minerals recovered prior to the extraction and/or from the alcoholic extract are collected and form a further product of the process according to the invention.

As pointed out above, the lipoidic fraction is not uniform and consists of a mixture of various materials which mxture is utilizable as such, e.g. as feed additive for poultry. The exact composition of the mixture will depend on the composition of the starting material, i.e. on the crop, and also on whether juicy or dry material serves for extraction with alcohol. As a rule, the lipoidic fraction amounts to 0.08—0.25% by weight of the original starting material and contains 6.2—15.2% by weight of chlorophyll, 0.5—2.6% by weight of carotenoids, 50—75% by weight of lipoids and 0.10—0.25% by weight of β-carotene. Where dry material is used for extraction the lipoidic fraction also contains certain amounts of minerals and proteins.

The yield in saponins depends on the age of the leaves as well as some other inherent factors. It amounts as a rule to 0.07 to 0.7% by weight of the dry flesh or 0.1—1% by weight of the starting juice, or 0.5—6% by weight of the slurry obtained, as described above, by letting the juice stand and removing the supernatant clear solution.

It is self understood that any solvents used in the course of the process according to the present invention and removed at a subsequent stage by evaporation may be recovered and recycled.

The invention is illustrated by following Examples without being limited thereto.

EXAMPLE 1.

10 litres of juice expressed from agave leaves was adjusted with milk of lime to *p*H 7.8 and heated up to boiling temperature. The precipitate formed was separated by decantation of the supernatant mother liquor, strained through a cloth and then dried at 60° C. in vacuo. There was obtained 385 g. of a bright green powder containing *inter alia*, 19.85% of minerals as ash, 10.5% of crude protein, 8.76% of calcium oxide, 0.58% of magnesium oxide, 0.165% of chlorophyll, 0.060% of carotenoids, 0.004% of β-carotene, 2.86% of lipoids, 0.15% of phosphorus.

The dark green mother liquor was concentrated in vacuo at 60° C. until a water content of about 45%. The thick mass obtained congealed at 10° C. to yield 1640 g. of gel. 2410 ml. of 98% methanol was then added to the gel, and the mixture thus obtained boiled under reflux and filtered through a cloth while hot. The filter residue was again extracted with two portions of 75% methanol of 2 litres each. The extracts were combined, cooled to 30° C. and re-extracted with petroleum ether (40—50° C, fraction), until the green colouring matter had completely passed into the petroleum ether phase. The petroleum ether layer was concentrated to a volume of 100 ml. and extracted with three 1.5 litre portions of water to remove glycosides and other water-soluble substances. The petroleum ether layer was then evaporated to dryness whereby 11 g. of a thick green mass was obtained, containing 56.45% of lipids, 6.06% of chlorophyll, 1.16% of carotenoids, 0.0936% of β-carotene.

The combined alcoholic extract was divided into three equal portions.

(*a*) *Isolation of crude saponins.*

The first portion of the alcohol extract was acidified to *p*H5 and heated to boiling. The precipitate formed was filtered off, cooled, washed with 0.5 litres of 90% methanol. The methanolic washing was combined with the original extract and the combined solution evaporated to dryness, whereupon a yellowish-brown mass of crude saponins was obtained. After crushing, 81.5 g. of a hygroscopic powder was obtained, having a foam number of 6,000.

(*b*) *Isolation of purified saponins.*

The second portion of the alcoholic extract was concentrated by distilling off the entire alcohol. The residual aqueous layer was mixed with 4 litres of *n*-butanol, acidified with sulphuric acid to *p*H2, and the aqueous-butanolic azeotrope was distilled off. The hot anhydrous butanolic saponin solution remaining in the flask was decanted off the precipitate adhering to the bottom of the flask which contained i.e. polysaccharides, and after cooling, mixed with acetone in the ratio 1 : 2. The precipitate was isolated by decantation of mother liquor as far as possible followed by filtration of the remaining solution through linen. The saponin so isolated was washed quickly with 0.2 litre of acetone and dried at 80° C. in vacuo. 31.0 g. saponins were obtained; foam number 16,000.

(*c*) *Isolation of saponins and sapogenins.*

The third part of the alcoholic extract was concentrated by distilling off the entire alcohol, and the solution thus obtained was kept in the refrigerator at +5° for 24 hours. The precipitate formed was centrifuged off, washed with ice water, then dried at 80° C. in vacuo. During the drying, a strong foaming occurred. As a result, 14.5 g. of light brown foamy mass of saponins was obtained; foam number 10,000. From the filtrate 2.6 g. of crystalline hecogenin was obtained by acid hydrolysis and working up as known per se.

EXAMPLE 2.

5 litres of juice expressed from agave leaves was adjusted to *p*H 7.8 by the addition of milk of lime and thereafter the water was evaporated in vacuo to dryness at about 60° C. The residual powder which amounted to 725 g. was ground in a mortar. 400 g. of the ground powder was admixed with 2 litres of *n*-butanol, and the mixture refluxed. After separation of the extract, extraction was repeated with another 2 litre portion of butanol. The extracts thus obtained were combined, cooled to 20° C. and the precipitate formed (mainly glysocides) filtered off and washed with butanol. The combined butanol extracts were then concentrated to about 0.5 litre and a second crop of glysocides which precipitated was filtered off. The precipitate was washed with 0.25 litres of butanol and the washing added to the solution. The final solution consisting of extract and washings was mixed with petroleum ether (40—60° fraction) in the ratio 1 : 2 whereupon a further precipitate was obtained, consisting of the remainder of the glysocides. The precipitate was filtered off and the filtrate mixed, while stirring, with 360 ml. of distilled water. Two layers formed which were separated from each other. The lower, water-butanol layer was re-extracted with two portions of petroleum ether and the resulting extract was combined

with the first petroleum ether extract. The combined petroleum ether extracts were evaporated in vacuo at 60° C., whereby 6.92 g. of a thick green substance was obtained containing 5.2% of chlorophyll, 0.6% of carotenoids, 0.102% of β-carotene, 78.6% of lipids.

The combined glucoside precipitates were dried in vacuo, yielding a yellowish-brown powder amounting to 56.2 g. (about 2% by weight of the starting juice). The precipitate was dissolved in anhydrous methanol, the solution acidified with sulfuric acid to *p*H2, filtered and the saponins precipitated by an addition of acetone. 40.3 g. of saponins were obtained, having a foam number of 16,000.

The remaining 325 g. of the dried material which remained after evaporation of the juice to dryness were extracted in the heat twice with a methyl alcohol-petroleum ether mixture (2 : 1), and then three times with 99% methanol acidified with sulphuric acid, using 2 litres of solvent each time. The amount of sulphuric acid was so chosen, that a sample of the solution, when diluted with an equal volume of water, showed a *p*H of 2. The *p*H of the combined extracts obtained was then raised to 4 by the addition of a 50% aqueous sodium hydroxide solution. The mixture was left to stand, and the alcoholic and petroleum-ether layers were then separated. The alcoholic layer was re-extracted twice more with petroleum ether, the petroleum ether solutions combined and evaporated to dryness. A thick green mass was obtained (2.1 g.) containing 4.8% of chlorophyll, 0.5% of carotenoids and 0.081% of β-carotene. The alcoholic extract was worked up for the recovery of hecogenin as known per se. 5.2 g. of pale cream coloured hecogenin (m.p. 248° C.) were obtained.

EXAMPLE 3.

Juice from agave sisalana was adjusted to *p*H 8.0. 4 litres of this juice was concentrated in vacuo at 60—70° C., yielding 1028 g. of a thick mass, containing about 50% of water (dry matter 518 g., water 510 g.). The mass was then extracted with *n*-butanol under reflux at the boiling temperature of the azeotropic butanol/water mixture. The extraction was repeated four times, using 12 litres of butanol for each extraction. After each extraction the liquid phase was separated by straining through a cloth. The butanol extracts thus obtained were freed of sugars by distilling off the azeotrope and filtering while hot. After cooling, the glycosides precipitated and were removed by filtration. Thereafter the solution was concentrated to a volume of 0.5 litres, and after cooling, a second crop of glysocides was filtered off. The glycosides precipitate were transferred to a vessel containing 1.5 litres of petroleum ether and 0.25 litres of butanol, the contents of the vessel thoroughly mixed, and the undissolved matter filtered off and washed with 0.5 litres of petroleum ether. The petroleum ether-butanol filtrate was mixed with 0.5 litres of the butanolic mother liquor from the second crop of glycosides whereupon there precipitated a third crop of glycosides which was filtered off and washed with 0.5 litre of petroleum ether. The petroleum ether from the washing was added to the mother liquor and the combined solution concentrated to a volume of about 200 ml. Thereafter 0.5 litre of petroleum ether was added to the concentrated solution and subsequently 100 ml. of distilled water was added while stirring. Stirring was continued for some time and the mixture was then left to stand at room temperature whereupon it separated into an upper petroleum ether layer and a lower butanol/water layer. The two layers were separated, the butanol/water layer washed twice with 0.5 litre of petroleum ether, the resulting extract added to the petroleum ether solution which in its turn was washed with 0.5 litre of water. From the washed petroleum ether solution the solvent was evaporated to dryness which yielded 10.40 g. of a thick, green mass containing *inter alia*, 70.2% of lipids, 10.87% of chlorophyll, 2.05% of carotenoids, 0.25% of β-carotene, and about 0.25% of unidentified other substances.

The combined glycoside precipitates were dissolved in 2 litres of anhydrous methanol acidified to *p*H2 and precipitated by the addition of 4 litres of petroleum ether. As phase separation occurred, 2 litres of acetone were added. The precipitated saponins were filtered off, quickly washed with acetone and dried in vacuo. 38.6 g. of a yellow powder was obtained; foam number 20,000.

EXAMPLE 4.

15 litres of juice, expressed from 6—8 year-old agave leaves, were left to stand for five days at a temperature varying between 35° C. at daytime to 18° C. at night. After five days 11 litres of clean supernatant solution were removed while the remaining 4 litres of bottom suspension were taken as starting material.

One litre of this suspension was thoroughly agitated and admixed with 4 litres of 99% methanol. The mixture was refluxed for half an hour. The undissolved matter was filtered off and washed with ½ litre of hot 99% methanol.

The filtrate was cooled, the precipitate formed filtered off again, and washed with ½ litre of 80% methanol. 1.5 g. of precipitate, almost white in colour, was obtained, which after hydrolysis with 3N sulphuric acid and

purification yielded 1.217 g. of pure hecogenin.

The alcoholic filtrate was extracted with light petroleum ether for the removal of pigments. The petroleum ether was distilled off leaving behind 6.6 g. of a green mass containing 83.4% of lipids, 3.6% of chlorophyll and 0.56% of carotenoids.

From the aqueous-alcoholic solution, remaining after the extraction of lipids, the alcohol was distilled off and the aqueous solution obtained was cooled. A precipitate formed which was centrifuged off, washed with cold water and dried, yielding 18.5 g. of saponins of foam number 10,000. The precipitate was not fully soluble in water.

The filtrate was boiled with sulphuric acid (final concentration 3N). A precipitate formed from which 1.31 g. of pure hecogenin was obtained.

The saponins obtained were hydrolysed, yielding additional 3.345 g. of hecogenin.

Another litre of the above suspension was evaporated to dryness. The dry residue was extracted first with one litre of hot methanol, followed by three portions of 0.5 litre each. The extracts obtained were combined and concentrated to 0.8 litre and mixed cold with 2.5 litres of acetone. The saponin precipitate obtained was filtered off, washed with acetone and dried in vacuo. 21.3 g. of saponins of foam number 10,600 was obtained.

The acetone-alcoholic mother liquor was concentrated to 0.5 litre, 15% of water was added to the concentrate and the mixture extracted three times with 0.5 litre of petroleum ether (60—80° C.). The petroleum ether extracts were combined and the petroleum ether evaporated which yielded 6.1 g. of the lipoid fraction containing 82.3% lipids, 2.4% of chlorophyll and 0.51% of carotenoids. The raffinate remaining after the lipoid extraction was diluted with an equal volume of water. A precipitate formed which was filtered off and washed with 50% methanol. 1.21 g. 2 of practically white precipitate were obtained, which was boiled with 15% sulphurc acid for four hours. The product was then isolated by extraction and crystallization and consisted of 0.91 g. of pure hecogenin.

EXAMPLE 5.

26 litres of juice were expressed from the leaves of 9-year old agave.

4 litres of the juice thus obtained were neutralised to *p*H7 with borax and then concentrated, yielding 870 g. of a thick mass containing 525 g. of dry matter.

The mass obtained was admixed with 1.9 litres of hot 98% methanol. The undissolved matter was filtered off and the extraction repeated twice with 1-litre portions of hot 85% methanol. The combined residue was dried, yielding 278 g. of white powder, consisting mainly of mineral matter.

The combined extracts were re-extracted four times with 1-litre portions of petroleum ether (80—100° C.). The petroleum ether extracts were combined and upon evaporation of the solvent 8.3 g. of a lipoid fraction was obtained, containing 2.65% of chlorophyll, 0.61% of carotenoids and 82.5% fats.

Another 4 litres of the juice were left to stand for 10 days, and thereafter one litre of bottom suspension was separated, neutralized to *p*H7 with borax and evaporated to dryness in vacuo. 181 g. of a light grey-green substance was obtained, which was boiled thrice with 0.75 litre of absolute methanol. Thereafter the dry matter was again extracted twice with methanol acidified with sulphuric acid to *p*H1. The extracts obtained were combined, cooled and admixed with 10 litres of acetone. The precipitate formed was filtered off, rinsed with acetone and dried in vacuo. 37.5 g. of a light yellow, hygroscopic saponin powder was obtained. The filtrate was neutralized to *p*H7 with caustic soda and concentrated to a volume of one litre. The solution thus obtained was admixed with 150 ml. of water and extracted with petroleum ether (60—80° C.), the petroleum ether was separated and the solvent evaporated. In this manner 6.3 g. of a lipoid fraction was obtained.

The aqueous-alcoholic extract was diluted with 850 ml. of water and the resulting precipitate filtered off. 5.03 g. of precipitate was obtained which upon hydrolysis with 20% sulphuric acid and purification yielded 2.3 g. of pure hecogenin.

WHAT WE CLAIM IS:—

1. Process for the recovery of saponins and sapogenins from the non-fibrous matter or agave leaves and simultaneous recovery of non-saponinic organic and mineral matter generally referred to herein as "lipoidic fraction", comprising producing from said non-fibrous matter an extract with an aliphatic alcohol having not more than 6 carbon atoms in its molecule or a mixture of organic solvents containing at least one such alcohol, recovering at least a part of the lipoidic fraction from the alcohol extract and thereafter recovering saponins and sapogenins from this alcoholic extract.

2. Process according to Claim 1, wherein juice expressed from the agave leaves serves as starting material, comprising the steps of concentrating said juice down to a dry matter content of 50—70% and submitting the remaining syrup to extraction with an alcohol.

3. Process according to Claim 2, wherein any lipoidic fraction precipitating during concentration of the juice is separated.

4. Process according to Claim 1, wherein the juice expressed from the agave leaves serves as starting material, comprising the step of allowing the juice to separate into a slurry and supernatant clear solution, removing the latter and using the former for further processing.

5. Process according to Claim 4, wherein said slurry is further concentrated.

6. Process according to Claim 1, wherein dry matter obtained by drying of the juice and/or the flesh of the leaves is used as starting material for the extraction.

7. Process according to Claim 6, wherein the extractant is an azeotropic mixture of benzene and methanol.

8. Process according to any one of the preceding claims, wherein the removal of at least part of the lipoidic fraction from the alcoholic extract is effected by back extraction of the extract with a non-polar organic solvent followed, if desired, by recovery of the lipoidic fraction so back extracted.

9. Process according to any one of the preceding claims, wherein the removal of at least part of the lipoidic fraction from the alcoholic extract is effected by filtration and/or centrifugation of the extract.

10. Process according to any one of the preceding claims, comprising the step of recovering from said alcoholic extract any lipoidic fraction remaining after the recovery of the saponns and sapogenins.

11. Process for the recovery of saponins, sapogenins and lipoidic fraction (as herein defined) from the non-fibrous component of the agave leaves, substantially as hereinbefore described with reference to the Examples.

12. Saponins, sapogenins and lipoidic fraction (as herein defined) when obtained by the process according to any one of Claims 1 to 11.

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